SYNOPSIS

Synthesis, Characterization and Applications of Zinc, Copper and Silver Nanoparticles: A Green Approach

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1. Introduction

On the earth, there is the presence of material with different size and shape possessing different characteristics with varied properties and applications. Particles within the size range of 1-100 nm are considered as nanoparticles (NPs) [1] and the tailoring of material at their atomic level to attain the unique properties, which can be suitably manipulated for the desired applications, is the study of a well-known field called nanotechnology. The concept of nanotechnology as volcanic activity, combustion (*metalbhasam*), food cooking and vehicle exhausts was not new in the world, but understanding and examination have been done after 1974 [2]. NPs receive attention for their positive impact in improving consumer products, cosmetics, pharmaceuticals, antimicrobial agents, energy, transportation, agriculture, etc. [3]. In addition, NPs are important as they bridge the gap between bulk materials and its atomic or molecular structures [4].

Prof. Norio Taniguchi first introduced the concept of nanotechnology in 1974 with the multidisciplinary discipline covering research and technology from physics, chemistry and biology [2]. After this in 1989, a research article "Nanocrystalline Materials" reported by Gliter [5] has been proved a milestone in the way of progress to research in field of NPs. This article has more than 1300 citations since its publication which shows its importance in the field of nanotechnology [6]. In the past three decades, society has been explored more towards the nano-enable products. The estimated global production of Metal NPs in 2010 was of 260,000–309,000 metric tons. At the top of the list are: silica, titanium, aluminium, iron and zinc oxides NPs [7]. Detailed studies of NPs in field of industrial applications have been reported in various publications and research articles [8-9].

1.1 Properties of Nanoparticles

Particles in the nano range are of great interest and importance due to their extremely small size and having large surface area to volume ratio, which lead to both chemical and physical differences in their properties [10-11]. The field of Nanotechnology is growing day by day due to the increased applications of NPs in the routine of human life in medicine, agriculture, clothing, cosmetics, food processing, electronics, etc. [12-15]. Moreover, the properties of NPs highly depend on their shape and size [16].

1.1.1 Zn NPs

Zinc is a transition metal with atomic number 30 and is 24th most abundant element present in the earth crust. Zinc possesses two types of crystalline structure, Wurtzite and Zinc blende. However, it mainly exists in the Wurtzite form [17]. Zinc NPs are known for their low cost and properties like sensing, degradation of dyes, antimicrobial activity etc. [18]. The important application of Zn NPs is to absorb a wide spectrum of radiation and hence used for manufacturing cosmetic creams which protect from harm-full ultra violet radiations [20]. Moreover, photocatalytic and biomedical applications such as biomedical imaging, drug delivery, gene delivery, and biosensing are of great interest in case of ZnO NPs [18-20].

1.1.2 Cu NPs

Copper is a transition metal with atomic number 29. Important properties of Cu are its antibacterial activity and charge conduction capacity in the electronic world [3, 21]. Cu NPs are particularly more investigated because of their high natural abundance, low cost and good conducting properties. Cu NPs also show potential applications in sensing, photo and electro catalysis etc. [16]. Cu NPs are used in agriculture, consumer products, cosmetics, transportation, and pharmaceuticals as antimicrobial agent [3].

1.1.3 Ag NPs

Silver is a transition element with atomic number 47. Ag is very precious element; despite of this a lot of work has been done on the Ag NPs. This is due to its wide range of applications either in the optoelectronics or in the biological system related to the health issues. Ag is commonly used in drug delivery, drug imaging and also as a drug itself [1]. Ag NPs are effective against broad-spectrum bacteria, having antimicrobial and antioxidant properties. Moreover, Ag has been used to prevent HIV from binding to host cells and to provide relief from the mental stresses [1, 22]. In ancient time, silver was used as storage device and silver nitrate solution was directly used for wound healing during “Second World War” [23].

1.2 Synthesis of Nanoparticles

Numbers of methods have been developed for tailoring the NPs. These are classified as: chemical, physical and biological or green methods. Chemical synthesis methods involve the microwave, photochemical and chemical reduction methods, etc. Physical methods involve pulse laser deposition, mechanical/ball milling and pulsed wire

discharge methods [2, 5, 9, 24]. In biological or green methods, plants or microorganisms such as bacteria, fungi, yeasts, algae have been used [24-25].

The biosynthesis of metal NPs is an innovative and expanding area of research as compared to chemical and physical methods of synthesis, due to its eco-friendly development and use of renewable resources. Chemical synthesis generally involves the use of costly and toxic chemicals, whereas biological methods provide us a reproducible, economic, nontoxic and energy efficient alternative for large scale production of NPs [25]. The mechanism behind the reduction of metal salts in the biological synthesis is considered as the role of various chemicals like enzymes, proteins, amino acids, vitamins, polysaccharides and organic acids such as citrates or the phytochemicals present in the bio-material or plant extracts, which act both as reducing and capping agents [3, 26].

Comparing the biological identities i.e. micro-organisms like bacteria, fungi, yeasts, algae and plants for their efficiency in the NPs synthesis, plants are relatively straightforward and advantageous approach than micro-organisms. The use of micro-organisms require some special, complex and multi-step procedures such as isolation, culture preparation and culture maintenance, where as plant material can easily be collected, stored and used for synthesis purpose [27-29]. Therefore, biological methods which use plant extracts for synthesis of NPs are being investigated extensively.

1.2.1 Selection of plants

Biological methods are being advanced by changing the plant or its part for the preparation of extract, pH, temperature and concentration of precursors. The NPs shape, size and structure varies with these parameters, consequently, selection of plant plays a crucial role [26]. Therefore, in the present work, we have selected the leaves of plants"*Aloe Barbadensis* (Aloe Vera)" [30] and "*Ocimum Tenuiflorum* (Tulsi)" [31] for synthesis of Zn, Cu and Ag NPs as these are easily available and have many medicinal applications.

1.2.1.1 Aloe Vera

Aloe Vera is as short-stemmed or stemless plant **(Fig. 1)** growing to 60-100 cm in height. The leaves are thick and fleshy, green to grey-green in color and spread around the short stem. The leaves hold a translucent gel, extremely bitter in taste and known for their healing properties and other important applications [30]. The composition of translucent gel is of around 96% water, some organic and inorganic

compounds, eighteen out of twenty amino acids found in the body, phospholipids and some vitamins. Overall Aloe Vera consists of 75 constituents in the form of vitamins, enzymes, minerals, sugars, lignin, saponins, salicylic acids and amino acidswhich may be considered as the constituents that take part in the formation of NPs from the metal salts [32]. Aloe Vera is being used widely in medicine since ancient time due to its healing, skin and body anti-aging, antiviral, antitumor, anti-inflammatory and anti-diabetic properties [33-35].

Fig. 1 Aloe Vera Plant

Fig. 2 Tulsi Plant

1.2.1.2 Tulsi

Tulsi is an erected plant **(Fig. 2)** attaining a height of about 20-50 cm [31]. Different parts of Tulsi are used in *Ayurveda* as medicine for prevention and cure of many illnesses like cough, influenza, cold, headache, fever, colic pain, bronchitis, asthma, hepatic diseases, fatigue, skin diseases, arthritis and digestive disorders. Consequently, Tulsi is described as *"Queen of Herbs"* from the ancient time.The main constituents of Tulsi are alkaloids, glycosides, tannins, saponins and some aromatic compounds which may be considered as the constituents that take part in the reduction of metal salts to corresponding NPs [36].

2. Literature Review

The use of metallic NPs seems to be started with the beginning of glass-making in Egypt and Mesopotamia in the thirteenth and fourteenth centuries. Scientists have analyzed lot of antiquities, for which the unusual colors have been attributed to the

presence of metallic NPs [37]. Numerous researches have been reported on synthesis and applications of the metal oxide NPs. NPs are playing crucial roles with advanced applications [38] and superior properties like electrical conductivity, mechanical strength, magnetic properties, and thermal ability etc. than bulk materials [39]. For example, instead bulk materials, ZnO NPs are being used to eliminate impurities of sulphur and arsenic from contaminated water [40]. As both, industrial as well as medical applications require purity with uniformity in shape and size of NPs, the transition metals NPs are being investigated more [41].

The two approaches, which are used for synthesis of NPs are top-down and bottom-up. The commonly applied approach is bottom-up approach, which applies chemical and biological methods as compared to top-down approach which generally involves the physical method [21]. Chemical methods mainly involve reduction through chemicals by employing solvothermal/hydrothermal technique, sol-gel method, polyol method, etc. [5, 9, 42]. However, chemical reactions are hazardous, costly, consuming high energy and difficult to scale up. Consequently, green method are developing as an alternate for NPs synthesis to make the reaction non-hazardous, ecofriendly, cheap, energy efficient and to enhance the stability of synthesized NPs [26, 43].

2.1 Zn NPs

Zinc Oxide has vast and potent properties like large binding energy, wide band gap and high piezoelectric property. It is used in large number of devices like laser, optoelectronic, electromagnetic coupled sensor and surface acoustic wave devices [40]. The worldwide annual production of ZnO NPs is estimated to be between 550 and 33,400 tons [38]. Zhang et al. [42] reported the controlled synthesis of ZnO NPs with the use of different solvents like water, heptane and ethanol, which results into formation of flower, snowflake and prickly sphere or rod like structures, respectively. Singh et al. [44] has reported the first plant extract synthesis of ZnO NPs in 2011. They used the latex of *Maddar* plant for the reduction of the Zinc acetate di-hydrate at pH 12 and reported the particle size between 5-40 nm with spherical shape. Thereafter, in 2013 numbers of research articles have been reported in literature on the green synthesis of ZnO NPs. Nagarajan et al. [45] used sea weeds of *caulerpapeltata*, red *hypneavalencia* and brown *sargassummyriocystum* to synthesize ZnO NPs with size 36-186 nm, which shows effective antibacterial properties against

Gram-positive and Gram-negative bacteria. Bhumi et al. [13] reduced the zinc acetate using c*atharanthusroseus* leaf that results in formation of NPs in range of 23-57 nm with good antibacterial activity.

In 2015, Elumalai et al. [18] studied the photocatalytic activity of v*itextrifolia* leaves synthesized ZnO NPs for the photo degradation of methylene blue dye and antimicrobial activity. In another study, Hassan et al. [46] used *corriandrumsativum* leaf extract to prepare ZnO NPs with size of 9-18 nm for the photocatalytic degradation of anthracene dye. Rana et al. [47] synthesized dye efficient ZnO NPs using fruits extract of *terminaliachebula* with particles size of 12 nm for the degradation of Rhodamine B (RhB) dye. Suresh et al. [48] reported green synthesis of multifunctional ZnO NPs by *artocarpusgomezianus* leaf extract through solution combustion route. Koli et al. [49] prepared anti-diabetic and anti-microbial active ZnO NPs using *cheilocostusspeciosus* plant extract. Patilet et al. [50] reported ZnO NPs synthesized using *limoniaacidissima* with spherical particle size 12-53 nm and found efficient against the *mycobacterium tuberculosis* growth.

2.2 Cu NPs

Kulkarni et al. [51] synthesized Cu NPs using *Eucalyptus sp.* plant leaves, with particle size 27.65-48.19 nm surrounded by proteins and metabolites such as phenolic acids, carboxylic acids and flavonoids functional groups from the plant extract. The CuO NPs synthesized with *calotropisgigantea* leaf extract were used as an electro-catalytic material in dye-sensitized solar cell [52]. *Ocimum sanctum* leaf extract can reduce copper ions into copper NPs within 8 to 10 minutes of reaction time. Thus, this method can be used for rapid and ecofriendly biosynthesis of stable copper NPs [21]. Maruthupandy et al. [53] reported the synthesis of CuO NPs with the particle size of 17 nm and used them for excellent sensing of metal ions, viz. Li^+ and Ag^+.

2.3 Ag NPs

A lot of literature is available on synthesis and applications of Ag NPs. The estimated global annual production of Ag NPs is 55 tons [38]. However, the improvement to obtain standardized results is still in progress and is very important area to work. Synthesis of Ag NPs is focused mainly on two methods: chemical and biological. Siddiqui et al. [54] synthesized the AgCl and AgO NPs in the range of 2-10 and 2-12 nm, respectively by using the chemical capping and precipitation methods. In biological methods, literature reported the synthesis of Ag NPs with number of plants, fungi, bacteria and algae [28-29, 55-57].

Recently, synthesis of Ag NPs by using plant extract is getting more attention in comparison to the chemical methods. Rout et al. [55] reported the synthesis of Ag NPs using Tulsi and investigated their antibacterial and antifungal activities. Behera et al. [56] reported the synthesis of Ag NPs by using ten medicinal plants, which can be used ineffective synthesis of drugs against bacterial and fungal diseases. Banerjee et al. [57] synthesized the spherical, triangular and cuboidal Ag NPs, which are potentially effective against antimicrobial and toxicity analysis. Medda et al. [58] used Aloe Vera and concluded that Ag NPs can be applied as better fungicide in near future. Saraswathi et al. [59] reported the antioxidant, antimicrobial and anti-inflammatory properties of biologically synthesized Ag NPs.

3. Research Gap Identification

Although large numbers of synthesis methods are reported for the silver, zinc, and copper NPs, the literature shows that biological or green methods are relatively new and is an innovative approach. Moreover, these methods need to be elaborated and standardized for uniform shape and size of NPs. In addition to this, applications of biologically synthesized NPs for photocatalytic degradation of dyes and anti-microbial activities need much more attention.

4. Objectives

In the light of above discussions the objectives of present study are as follows:

1. To synthesize Zn, Cu and Ag NPs by elaborating and standardizing the existing green methods of synthesis.
2. To characterize the synthesized NPs by using different characterization techniques like XRD, SEM, TEM, UV-Visible, etc.
3. To investigate the photocatalytic and antimicrobial properties of synthesised NPs.

5. Proposed Methodology

In the literature, numbers of chemical and biological methods of synthesis [4-9, 14-16] are reported. In present study, we propose to use the biological methods [25, 36-38], which involves the use of plant extracts of Aloe Vera/Tulsi for synthesis of NPs.

5.1 Materials

Zinc acetate, copper acetate, silver nitrate, sodium hydroxide, potassium hydroxide and ethanol of high purity will be used. Plant leaves of Aloe Vera and Tulsi will be collected from nearby areas for preparation of extracts.

5.2 Methods

5.2.1 Preparation of plant extract

Green leaves of Aloe Vera/ Tulsi will be collected and washes 2-3 times with normal tap water and then with distilled water to remove any short of impurities or contaminants. Then a fixed amount of washed leaves will be crushed and boiled in a fixed volume of water for said time. Boiled extract will be filtered by using whatman filter paper and the filtrate will be centrifuged to remove any short of solid or heavy particles present in the extract [44-50].

5.2.2 Synthesis of Nanoparticles

Metal salt solutions of required concentration will be prepared in the distilled water. A fixed volume of this solution will be taken in the round bottom flask and a fixed volume of above prepared plant extract will then be added dropwise to it with constant stirring and heating at standard parameters **(Fig. 3)** [44-50]. The reaction will be monitored with the help of color change of the reaction mixture.

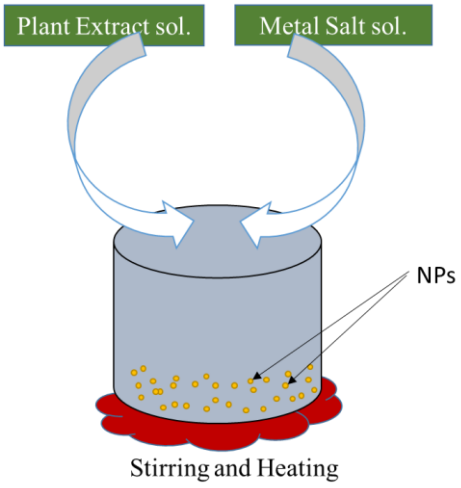

Fig. 3 Green Synthesis of Metal NPs

5.3 Characterization of Synthesized Nanoparticles

NPs are generally characterized by investing their size, shape and surface area. A homogeneity in these properties result in the advancement in applications of NPs. The characterization will be done by using UV–visible spectroscopy [60], scanning

electron microscopy (SEM), transmission electron microscopy (TEM), [61-63], X-ray diffraction (XRD) [21] and energy dispersive spectroscopy (EDS) [64].

5.4 Applications of NPs

5.4.1 Photocatalytic degradation of dyes

In this study, experiments will be performed to evaluate the photocatalytic properties of synthesized NPs by photocatalytic degradation of commercially available dyes. The photocatalytic decomposition of dyes will be examined by measuring the absorbance at regular time intervals by using UV-Visible spectrophotometer [65-67].

5.4.2 Antimicrobial activity

Synthesized NPs will be studied for their antimicrobial activity by investigating their inhibition zone/area for the microbes [48-50, 56-58].

6. Thesis Outline

CHAPTER 1 (Introduction): This chapter will contain the brief introduction regarding NPs, biological methods, plants extract and NPs of transition metal.

CHAPTER 2 (Literature Review): This chapter will deal with intense literature survey of synthesis and applications of transition metal NPs.

CHAPTER 3 (Materials and Methods): It includes the experimental details on material used, biological methods of synthesis, characterization techniques and applications of synthesised NPs.

CHAPTER 4 (Results and Discussion): This chapter will include detailed discussion on results of present work which includes the key points and various aspects of synthesis, characterization and applications of transition metal (Zn, Cu and Ag) NPs.

CHAPTER 5 (Summary and Conclusions): This chapter will summarise and correlate all the above mentioned chapters in lucid manner.

7. Research Plan Schedule

Research Activity	Progress in Months											
	3	6	9	12	15	18	21	24	27	30	33	36
Course work and extensive literature survey or design of work	Aug 2016 to Jul 2017 →											
Research gap identification, development of methodology and submission of synopsis			Jan 2017 to Dec 2017 →									
Experimental Work and Publications					August 2017 to December 2018 →							
Thesis writing and Publications									Dec 2018 to Jul 2019 →			

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